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PREPARATION OF POLYSTYRENE MICROSPHERES FOR LASER
VELOCIMETRY IN WIND TUNNELS

Cecil E. Nichols, Jr.

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Langley Research Center
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INTRODUCTION

Laser Velocimetry (L/V) technology has made great strides in replacing intrusive devices for wind tunnel flow measurement. At present, Langley Research Center (LaRC) has several wind tunnels which have dedicated L/V's, including the 14- x 22-Foot and 16-Foot Transonic Wind Tunnels. The Achilles' Heel of the L/V has not been the L/V itself, but proper size seeding particles having known drag characteristics and a means of introducing these particles into the wind tunnel flow.

After experimenting with various seeding concepts, it was decided that for most Langley applications the use of solid particles suspended in ethanol and injected through a fluid nozzle was preferred. Research also wanted particles which are spherical and monodisperse as well as having a specific diameter, diameter being a function of the particular tunnel and laser characteristics.

Initially, atomized kerosene was used as a seeding material but was proven to be unsatisfactory due to low data rate. The use of kaolin particles suspended in ethanol was then tried resulting in adequate data rate at very low cost. Kaolin is, however, not spherical nor is it monodisperse; therefore, something better was clearly needed. In fact, monodisperse spherical polymeric particles in the desired diameters are commercially available and were used on a limited basis with excellent success in some small wind tunnels at Langley; however, the cost is prohibitive in large wind tunnels due to the quantity of particles required and hence was not used.

At this point, the author decided to investigate the feasibility of in-house preparation of polymeric particles. Initial experiments were directed toward emulsion polymerization using surfactants^{1,2} but it was quickly learned experimentally that monodispersity was difficult to achieve by this method. Emulsifier-free polymerization³ was then tried with excellent results. All formulations contained in this report were prepared using this method. Emulsifier-free polymerization can be carried out only at a relatively low concentration of solids, i.e., approximately 10 volume percent in water. When particles are referred to in this paper, solid particles suspended in water (as prepared) are meant.

The purpose of this paper is to present detailed instructions, procedures and formulations which are being used by the author to produce particles presently being used in Langley's Wind Tunnels. It is hoped that the information given is detailed and practical enough to allow the interested researcher to produce high quality low-cost particles.

APPARATUS

Referring to Figure 1, the apparatus consists of a 3-liter Pyrex reaction kettle having temperature controlled by a heating mantle and a cold finger condenser circulating tap water. This control is operated by a mercury thermoregulator which alternately calls for heating or cooling depending on the set temperature versus the sensed temperature. A condenser returns any vaporized reactants to the reaction vessel. A gas inlet adapter atop the condenser allows a nitrogen purge. A "home-made" stirring paddle (shown full-scale in Figure 2) insures sufficient agitation of the reactants. Shaft size is not important and can be sized to use whatever bushing is at hand.

Following is a list of catalog numbers for various components of the apparatus, keyed to Figure 1:

- (1) No. 6947, Pyrex Kettle w/4 neck cover, 3000 ml (Corning Glass Works)
- (2) Condenser, cold finger (Ace Glass Inc., No. 5950)
- (3) Mercury Thermoregulator (Precision Scientific, No. 62539)
- (4) Condenser (Ace Glass Inc., No. 5945)
- (5) Adapter, Gas Inlet (SGA Scientific, Inc., No. JA 7970)
- (6) Stirring Paddle ("home-made")
- (7) Armoured Heating Mantle, 4 Liter (Glass-Col, Catalog No. TM 580)

Equivalent components from other manufacturers are also acceptable. The manufacturer is mentioned here only to present an accurate record as to what was actually done in the course of the author's experimentation.

PROCEDURE

1. Select formulation from Table 1 for desired particle size.
2. Charge the reactor in the following order: water, magnesium sulfate electrolyte solution (if required), and styrene.
3. Bubble nitrogen gas through the above mixture for 40 minutes in order to purge all oxygen from the reactor (Approximately 0.5 liters/min. flow rate) using a gas dispersion tube (Pyrex, ASTM 170-220 or equivalent). Remove tube from the reactor after 40 minutes and place nitrogen line onto gas inlet adapter atop condenser, maintaining this nitrogen purge throughout the entire run.
4. Start agitator (150 RPM) and begin heating to 65°C.
5. When temperature stabilizes at 65°C, as evidenced by several cycles of the temperature controller, add potassium persulfate solution to the reactor via pipet insuring that the pipet tip is several inches below the liquid surface. This places the initiator well beneath the styrene layer on top and into the reaction zone in the water layer where the polymerization takes place. Run for 24 hours (beginning with addition of potassium persulfate).

6. At the end of the 24 hour period unplug temperature controller and stop agitation. After cooling for a few minutes, remove any sticky, rubbery material which may form a separate layer on the top with paper towels. Filter through 100 mesh cheese cloth into a clean storage container. Filtration removes any coagulum or sticky substance from the particles. Please note that some styrene may polymerize on the stirring blade. The polystyrene adheres to the blade quite well but can be removed by soaking in xylene overnight.
7. (OPTIONAL) If particles are to be stored longer than several months, it is advisable to place the sealed container of particles into a 65-70°C oven for 24 hours. This will minimize any chance for biological growth as the particles appear to be an ideal culture medium.

If you use Step 7, sample for weight percent solids after Step 6.

SAMPLE FOR WEIGHT PERCENT SOLIDS

Pipet approximately two to four ml of particles each into two small pre-weighed disposable aluminum sampling pans. Weigh the respective samples and place in a 65-70°C oven for several hours until dry. Re-weigh each pan and calculate the weight percent solids by using the following formula:

$$\frac{(w_f - w_t) 100}{(w_i - w_t)}$$

Key:

w/o	weight percent
w _f	weight after drying
w _i	weight before drying
w _t	tare weight

The w/o solids averaged 7.3 in the author's preparations.

DETERMINATION OF PARTICLE SIZE VIA LIGHT MICROSCOPY⁴

A Nikon Microphot -FX Microscope at 2000X was used to determine particle size. This was done with Polaroid Photography by comparing an NBS 474 AR Chromium Photomask Optical Linewidth Standard at 2000X with a photograph of the particles at the same magnification. One drop of the undiluted particles was placed on a microscope slide and smeared by slowly scraping with a cover slide held on one edge. After drying in a dessicator (about one hour) the slide was then photographed under the microscope. A properly prepared slide will have regular arrays of particles as shown in Figure 3. Measurements should be made from center to center of the longest straight array of particles possible being very careful to avoid any arrays that exhibit microcracks. If

microcracks are not avoided, erroneous measurements will be obtained. Since some microcracks are extremely difficult to discern, it is best to make as many measurements of different arrays as possible and then average the results. Obviously any measurements which are suspected of containing microcracks should be eliminated from the average. I have found this measurement technique to agree with Scanning Electron Microscope (SEM) measurements to within 0.1 micron which should be close enough for laser velocimetry work.

FORMULATIONS

Magnesium Sulfate and Potassium Persulfate solutions were made using deionized water of 16-18 megohm-centimeter purity. The 1 w/o solution is made by weighing out 10.0 grams of the respective chemical into a 1-liter volumetric flask and filling to the 1-liter mark with deionized water. Use of less pure water may result in a somewhat different particle size but should not affect monodispersity. In a similar manner, the 3 w/o solution is made by using 30.0 grams of the chemical rather than 10.0 grams, all other factors being the same. Note that in Table 1, some formulations use 3 w/o potassium persulfate in lieu of 1 w/o. Actually one should work as well as the other with proper adjustment of volumetric quantity but the 3 w/o is used due to an inadequacy in the setup. Step 4 under PROCEDURE calls for heating to 65°C. As this step occurs before addition of potassium persulfate, the total volume is such that the thermoregulator bulb does not touch the liquid and hence temperature control is not possible. By using 3 w/o potassium persulfate, the initial volume is large enough to allow submersion of the thermoregulator bulb. A slightly different setup would allow the thermo-regulator bulb to contact the liquid in which case 1 w/o could be used for all formulations.

For reasons not fully understood at present, small variations in particle size from the values given in Table 2 do occasionally occur, but these variations do not affect monodispersity. Increasing the amount of magnesium sulfate tends to give larger particles while a decrease results in somewhat smaller particles.

All chemicals used were obtained from Polysciences:

Magnesium Sulfate, ultra pure, Catalog No. 1623 ($\text{Mg SO}_4 \cdot 7 \text{ H}_2\text{O}$)

Potassium Persulfate, Catalog No. 1057

Styrene, Catalog No. 0660

Equivalent grades from other sources are obviously also acceptable. The supplier is mentioned here only to present an accurate record as to what was actually done in the course of the author's experimentation.

TABLE 1
FORMULATIONS FOR POLYSTYRENE LATEX, MONODISPERSE, SPHERICAL

	Particle Diameter, microns				
	<u>0.6</u>	<u>1.0</u>	<u>1.7</u>	<u>2.0</u>	<u>2.7</u>
Water (ml)	2489	2329	2369	2200	2339
Magnesium Sulfate (ml)	-0-	56	56	56	56
Concentration (w/o)	1	1	1	1	1
Styrene (ml)	265	265	265	265	263
Potassium Persulfate (ml)	46	150	110	278	139
Concentration (w/o)	3	3	3	1	1

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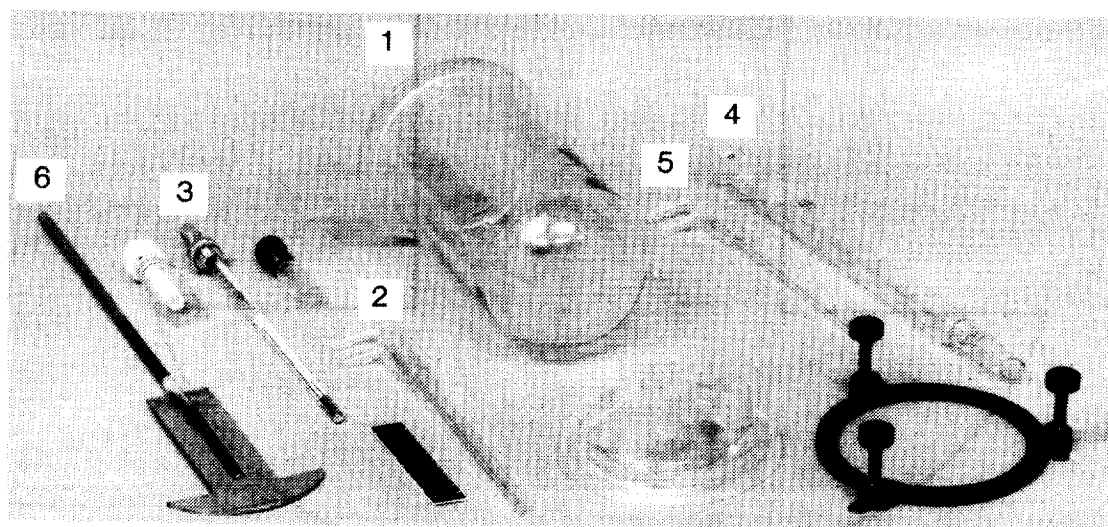
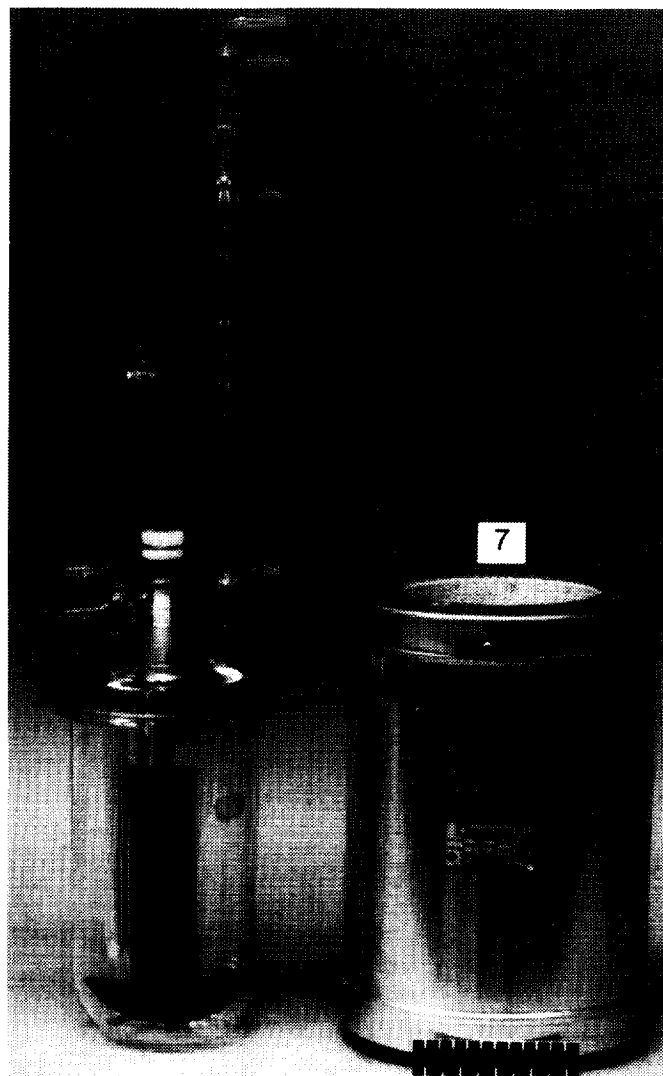
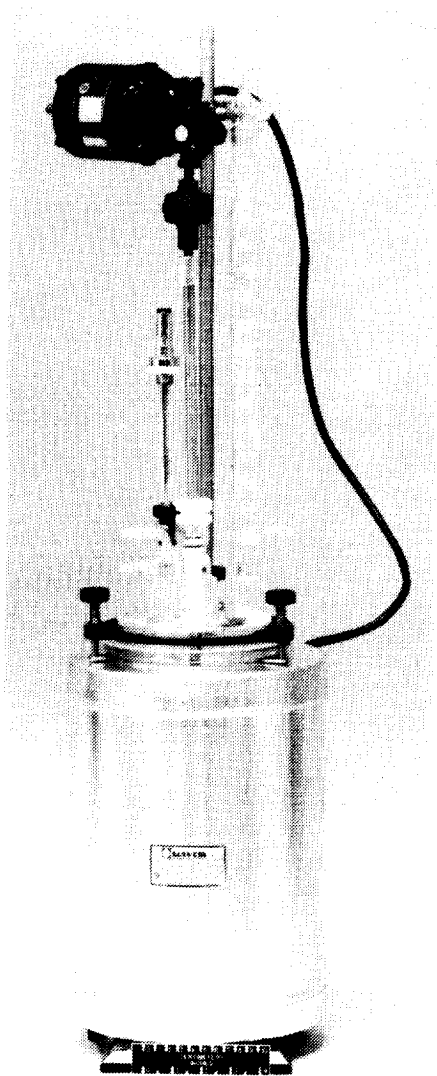


Figure 1 - Apparatus

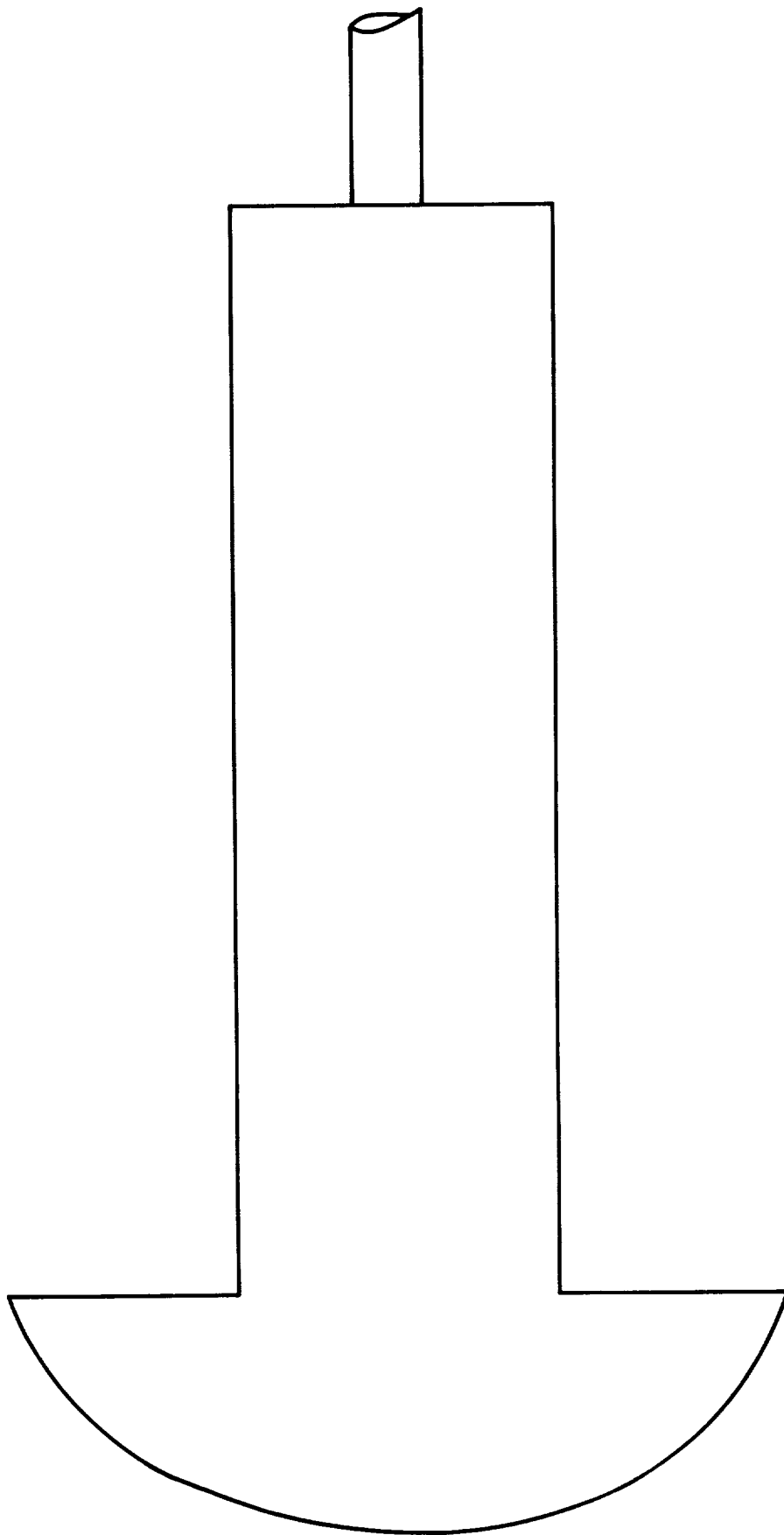
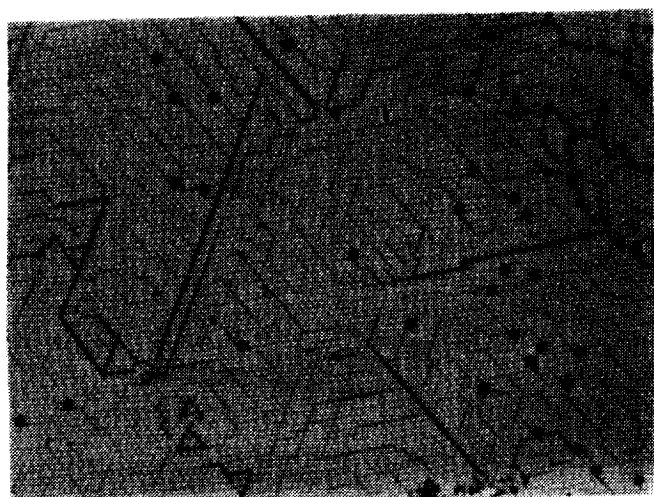
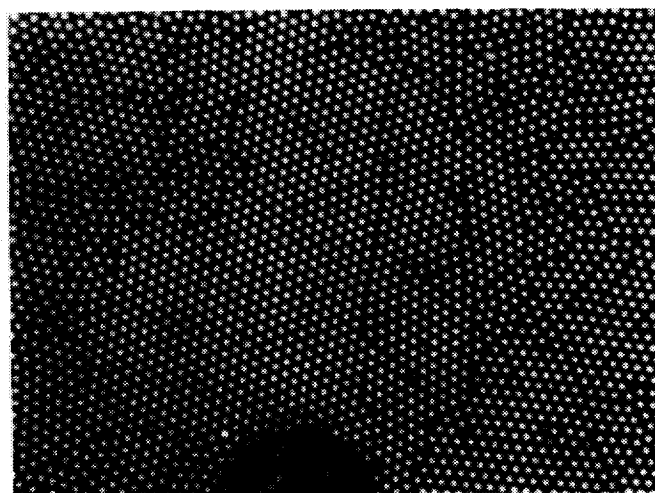


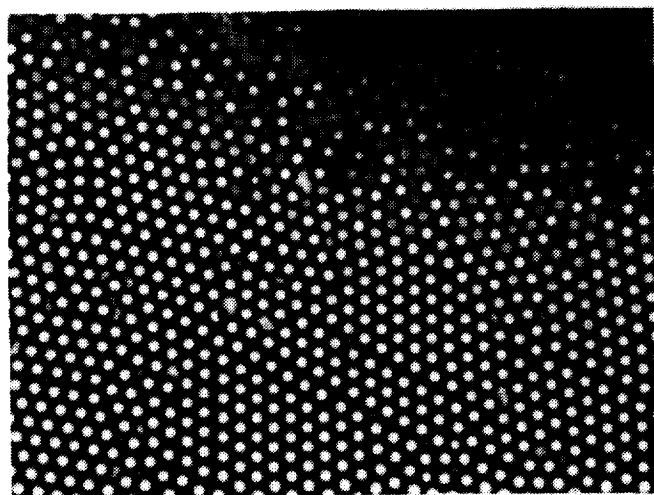
Figure 2 – Stirring Paddle (Full Scale)



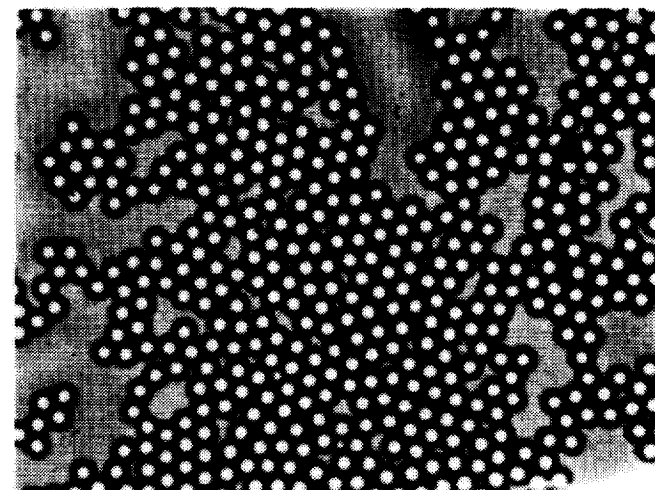
0.6 μ



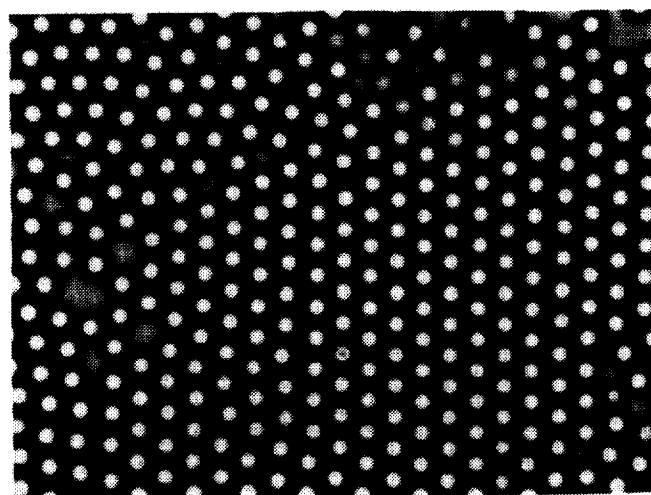
1.0 μ



1.7 μ



2.0 μ



2.7 μ

Figure 3 – Polystyrene Microspheres (2000x)

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